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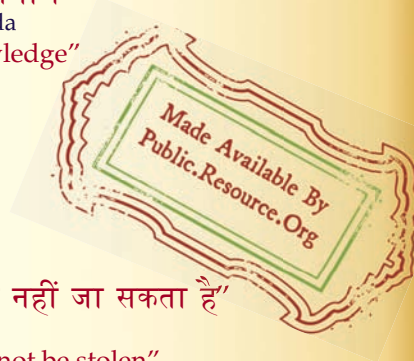
IS 11674 (1986): Method for determination of residual solvent in oilseed flours and meals by modified Pensky Martens closed tester [FAD 13: Oils and Oilseeds]



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“Knowledge is such a treasure which cannot be stolen”

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IS 11674 : 1986

“पुनर्विष्ट १९९४”

“RE-AFFIRMED 1994”

Indian Standard

**METHOD FOR DETERMINATION OF
RESIDUAL SOLVENT IN OILSEED FLOUR
AND MEALS BY MODIFIED
PENSKY-MARTENS CLOSED TESTER**

UDC 665.1/3.074.432 : 543.873.053

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 28 February 1986, after the draft finalized by the Nutrition Sectional Committee had been approved by the Agricultural and Food Products Division Council.

Residual solvent in oils and oilcakes could be a fire hazard and a source of toxicity. Differing quality of raw material, faulty plant operation, excessive through-put and varying steam pressure could result in excessive solvent concentrations in the final products. Accordingly limits of residual solvent are specified in the standard specifications.

The Pensky-Martens closed tester is used to determine flash point of fuels, lubricating oils and other viscous material; however its use with solid material like oilseed flours is not recommended. The modified Pensky-Martens tester as described in this standard is useful in determining flash points of oilseed flours and meals. Only two minor modifications are required: (a) A flat head on screw to plug the central hole, and (b) Provision in the cup of a central rod and a concentric ring both made of copper/brass to provide uniform heating.

This method is based on the work carried out at the National Physical Laboratory, New Delhi (India) and the apparatus as described in the Annual Book of ASTM Standards, 1985, Section 5, volume 05-03 Method E 134.

Indian Standard

METHOD FOR DETERMINATION OF RESIDUAL SOLVENT IN OILSEED FLOUR AND MEALS BY MODIFIED PENSKY-MARTENS CLOSED TESTER

1 SCOPE

1.1 This standard prescribes the method for determination of residual solvent in oilseed flour and meals by modified Pensky-Martens closed tester.

2 APPARATUS

2.1 A typical assembly of the apparatus, gas heated is shown in Fig. 1. The apparatus shall consist of a test cup, cover and stove conforming to the following requirements.

2.2 Cup

The cup shall be of brass or other non-rusting metal of equivalent heat conductivity and shall conform to the dimensional requirements given in Fig. 2. The flange shall be equipped with devices for locating the position of the cup in the stove. A handle attached to the flange of the cup is a desirable accessory. The handle should not be so heavy as to tip over the empty cup.

2.3 Cover

2.3.1 Cover Proper

The cover (Fig. 3) shall be of brass and shall have a rim projecting downwards, almost to the flange of the cup. The rim shall fit the outside of the cup with a clearance not exceeding 0.36 mm on the diameter. There shall be a locating or locking device or both, engaging with a corresponding device on the cup. The fire openings in the cover are shown in Fig. 3. The central hole receives a screw flush with the lower surface of the lid. In case of flash point operations for liquids, this hole receives a stirrer. The upper edge of the cup shall be in close contact with the inner face of the cover throughout its circumference.

2.3.2 Shutter

The cover shall be equipped with the brass shutter, approximately 2.4 mm thick, operating on the plane of the upper surface of the cover. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal centre of the cover between two stops, so placed that when in one extreme position, the openings A, B and C in the cover are completely closed, and when

in the other extreme position, these openings are completely opened. The mechanism operating the shutter shall be of the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme, the three cover openings shall be exactly open and the tip of the exposure tube shall be fully depressed.

2.3.3 Flame Exposure Device

The flame exposure device (Fig. 4) shall have a top with an opening 0.69 to 0.79 mm in diameter. This tip shall be made preferably of stainless steel, although it may be fabricated of other suitable metals. The flame exposure device shall be equipped with an operating mechanism which, when the shutter is in an 'open' position, depresses the tip so that the centre of the orifice is between the planes of the under and upper surfaces of the cover proper at a point on the radius passing through the centre of the larger opening A (Fig. 3).

2.3.4 Pilot Flame

A pilot flame shall be provided for automatic relighting of the exposure flame. A bead 4 mm in diameter may be mounted on the cover so that the size of the test flame can be regulated by comparison. The tip of the pilot flame shall have an opening the same size as the top of the flame exposure device (0.69 to 0.79 mm in diameter).

2.3.5 Device for Uniform Heating of the Flour/Meal (see Fig. 2)

This consists of a central rod (5 mm in diameter and 35 mm long), and a concentric ring (25 mm in diameter, 35 mm high, 1.0 mm thick) made of copper or brass. A portion (25 mm × 10 mm along the circumference) of the concentric ring is removed at one end to allow unhindered close contact between the meal and the thermometer bulb which enters the cup at an angle. The thermometer bulb should be 13 to 15 mm from the bottom. The central hole is plugged with a small screw, flush with the lower surface of the lid.

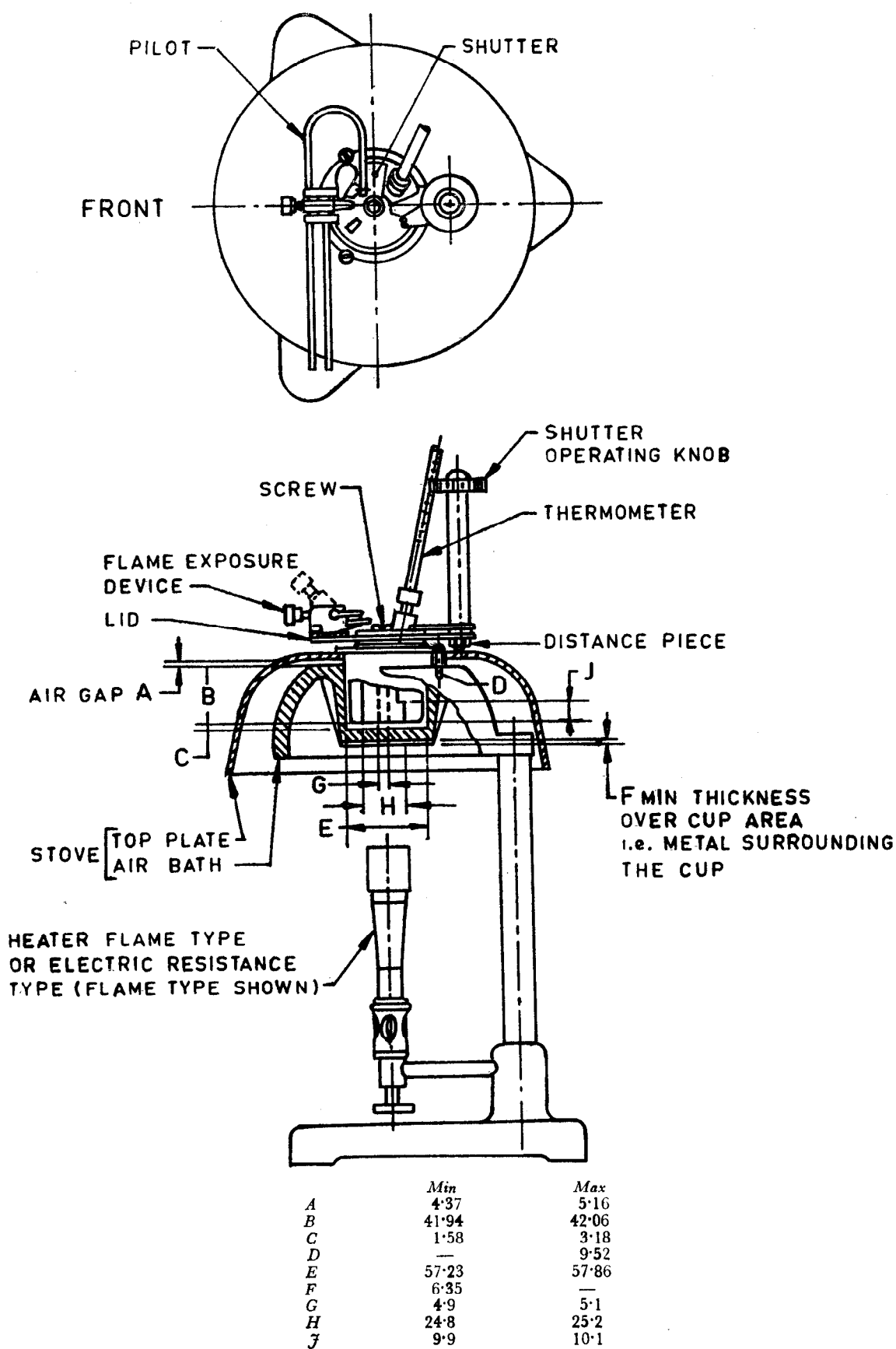
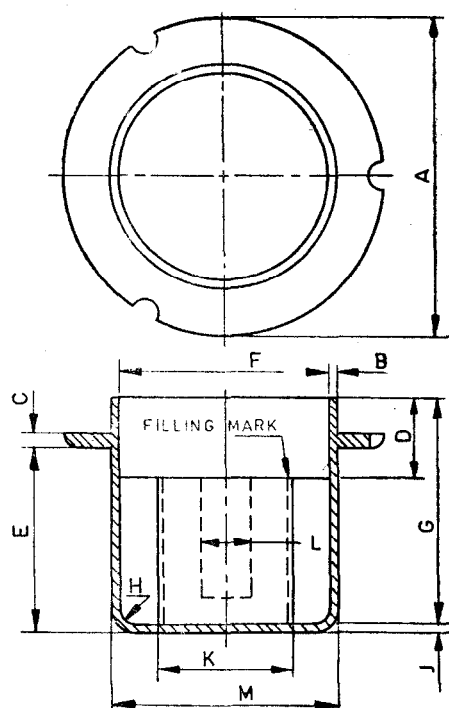


FIG. 1 A TYPICAL MODIFIED PENSKY-MARTENS CLOSED FLASH TESTER (GAS HEATED)



	Min	Max
A	79.0	79.8
B	1.0	—
C	2.8	3.6
D	21.72	21.84
E	45.47	40.72
F	50.72	50.85
G	55.75	56.00
H	3.8	4.0
J	2.29	2.54
K	24.8	25.2
L	4.9	5.1
M	53.90	54.02

All dimensions in millimetres.

FIG. 2 TEST CUP

2.4 Stove

Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. The stove shall consist of an air bath and a top plate on which the flange of the cup rests.

2.4.1 Air Bath

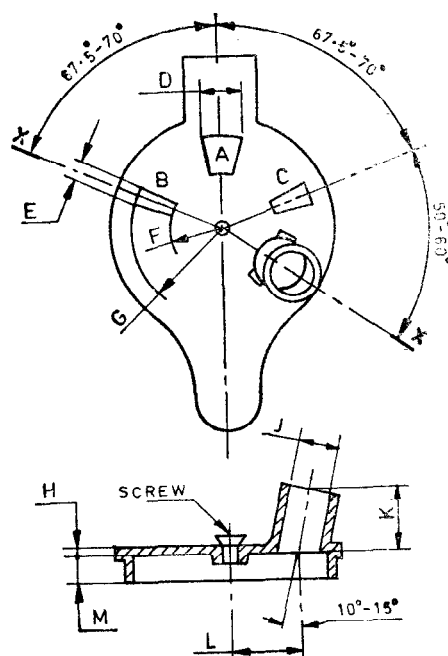
The air bath shall have a cylindrical interior and shall conform to the dimensional requirements as given in Fig. 1. The air bath may be either a flame or electrically heated metal casting (Note 1) or an electric resistance element (Note 2). In either case, the air bath must be suitable for use at the temperatures to which it will be subjected without deformation.

NOTES

1 If the heating element is a flame or electrically heated metal casting, it shall be so designed and used that the temperature of the bottom and the walls are approxi-

mately the same. On this account it should be not less than 6.4 mm in thickness. The casting shall be designed so that products of combustion on the flame cannot pass up and come into contacts with the cup.

2 If the air bath is of the electric resistance heated type it shall be constructed so that all parts of the interior surface are heated uniformly. The wall and bottom of the air bath shall be not less than 6.4 mm in thickness.



	Min	Max
D	12.7	13.5
E	4.8	5.6
F	13.5	14.3
G	23.8	24.6
H	1.2	2.0
J	12.27	12.32
K	16.38	16.64
L	18.65	19.45
M	7.9	—

All dimensions in millimetres.

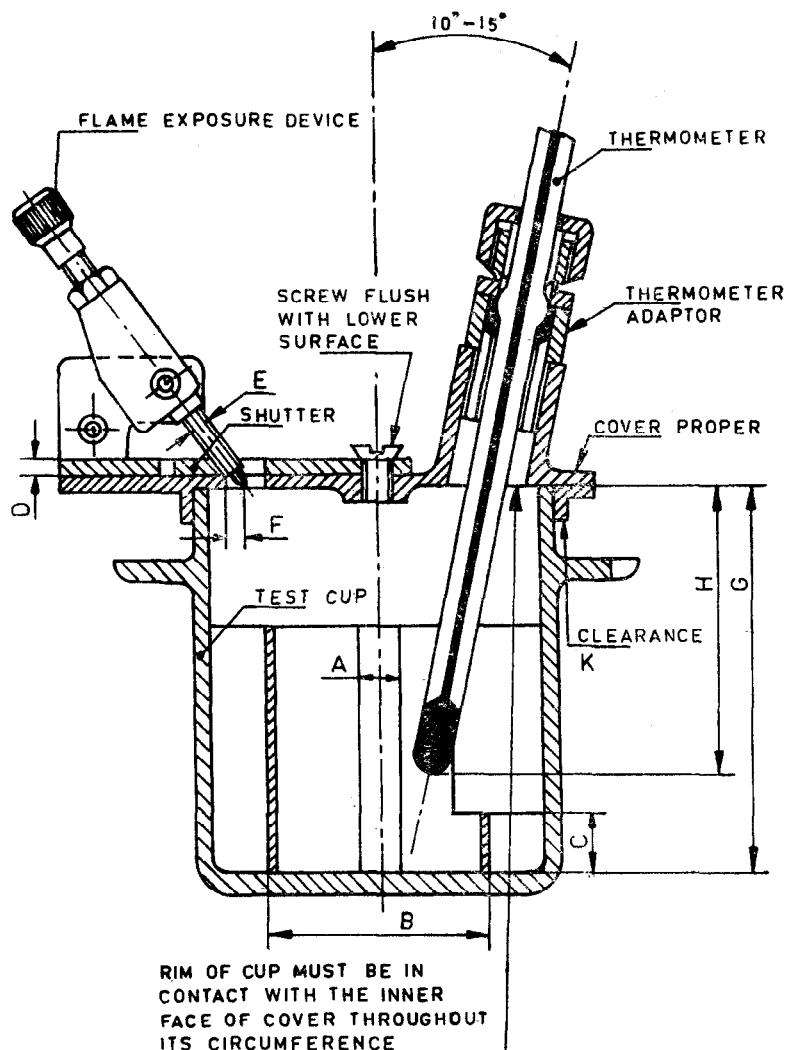
FIG. 3 COVER PROPER

2.4.2 Top Plate

The top plate shall be of metal and shall be mounted with an air gap between it and the air bath. It may be attached to the air bath by means of three screws and spacing bushings. The bushings shall be of proper thickness to define an air gap of 4.8 mm, and they shall be not more than 9.52 mm in diameter.

3 CALIBRATION

3.1 Take the oilseed kernels of the oilseed flour/meal to be tested and powder thoroughly and extract with hexane in a multipurpose solvent extractor or multipurpose automatic solvent extractor. Desolventize the extracted flour/meal under vacuum and then dry in an oven for many hours at 80°C. Keep the dried powder in a desiccator for use in calibration.



	Min	Max
A	4.9	5.1
B	24.8	25.2
C	9.9	10.1
D	2.0	2.8
E	0.69	0.79
F	2.0	2.8
G	55.75	56.00
H	43.0	46.0
K	—	0.36

All dimensions in millimetres.

FIG. 4 TEST CUP AND COVER ASSEMBLY

3.2 Take 100 g of the above powder. Put half of it in a 500-ml wide-mouthed empty bottle with a well-fitted screw cap carrying on its inside a piece of cork sheet covered with aluminium foil for air tightness. Add a known volume of hexane to the meal and shake the whole for 10 to 15 minutes and then keep for 1 hour to allow the solvent to permeate the powder thoroughly. Similarly prepare samples with 0.00, 0.01, 0.02, 0.05, 0.10, 0.20, 0.30, 0.50 and 1.00 ml of hexane. Cool each bottle in a refrigerator.

3.3 Fill the flour/meal powder tightly in the tester cup of the pre-cooled modified Pensky-Martens tester to the 35 mm mark as in standard testing. Any portion of the meal shall not be more than 15 mm from a copper heating surface of the cup or the concentric ring or rod. This results in uniform heating and the thermometer

shows a uniform rise in the temperature of the meal. About 30 g of the meal powder is necessary. Heat and maintain a rise of 1° per minute near the flash point while testing. Calibrate a curve for flash point against hexane concentration in the oilseed flour/meal.

4 PROCEDURE

4.1 Take about 30 g of the sample to be tested and carry out the procedure as given in 3.3. Determine the flash point and read the hexane in the sample from the curve as calibrated under 3.

NOTE — The flash point of flour/meal differs with its solvent content and its oil content. For proper evaluation of the residual solvent in the flour/meal, a reference sample of the particular oilseed containing same percentage of oil as the flour/meal under test should be taken for calibration.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices:

	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 331 01 31 331 13 75
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 86 62
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	2 18 43
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